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Status of Claims

Claims 1-6 are pending in the application. Claims 1 and 5 are allowed. Claims 2, 3, 4 and 6 are rejected under 35 U.S.C. §112, Second Paragraph. Applicants request that claims 3, 4 and 6 and certain instances in the specification be amended to correct a typographical error in Formula (Z'). No new matter has been added.

Rejection Under 35 U.S.C. §112, Second Paragraph

The Examiner rejected claims 2, 3, 4 and 6 under 35 U.S.C. §112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention. Specifically, the Examiner objected to the indefiniteness of the limitation "high-boiling point aprotic solvent" recited in Claims 2 and 4 which was believed to be undefined in the specification. Applicants respectfully traverse. The meaning and use of protic and aprotic solvents is well within the skill of organic chemists. To assist the Examiner, Applicants provide herein two references which define protic and aprotic solvents. The 4th Edition of Loudon's *Organic Chemistry* states that "A *protic* solvent consists of molecules that can act as hydrogen-bond donors. Water, alcohols, and carboxylic acids are examples of protic solvents. Solvents that cannot act as hydrogen bond donors are termed aprotic solvents. Ether, methylene chloride, and hexane are examples of aprotic solvents." Loudon, G. Mark, *Organic Chemistry*, 4th Edition, New York: Oxford University Press (2002) pg 317. Similarly, the 3rd Edition of the *Oxford Dictionary of Chemistry* states that "*Aprotic solvents* neither accept nor donate protons; tetrachloromethane (carbon tetrachloride) is an example." *Oxford Dictionary of Chemistry*, 3rd Edition, (1996) page 456. MPEP 2173.02 states that Examiners "should allow claims which define the patentable subject matter with a reasonable degree of particularity and distinctness. Some latitude in the manner of expression and the aptness of terms should be permitted even though the claim language is not as precise as the examiner might desire". In this respect, Applicants respectfully request that the rejection under 35 U.S.C. §112, second paragraph, be withdrawn.

Claims 3, 4 and 6 were also rejected under 35 U.S.C. §112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which

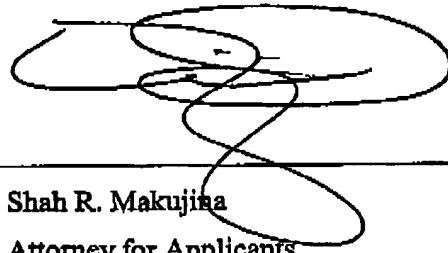
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applicant regards as the invention because as the Examiner correctly observed Z¹ in Formula (Z') had an incorrect valence due to a typographical error. Specifically, the error resided in having a double bond depicted between Y² and Z¹ instead of single bond. The correction of this error is unambiguous because Z¹ can only be C(O) which would require a single bond between Y² and Z¹. Moreover, one skilled in the art would recognize that Y² and Z¹ must be connected by a single bond in Formula (Z') since Applicants description of the process of preparing Formula (Z') depicts the cyclization of Formula (Y') which shows that Y² and Z¹ connected by a single bond. Applicants have duly corrected these typographical errors. The Examiner also objected to several schematic diagrams having intermediates with Y²=Z'-N, which appeared to have a problem with valence as well. Applicants have reviewed the schemes and have not found any errors in valency. The specific instances where Y²=Z'-N appears, Z' is attached to R⁷ which is bromo or chloro in which case the valency is correct. Therefore, Applicants respectfully submit that the rejection under 35 U.S.C. §112, second paragraph, be withdrawn.

In light of the amendments and remarks herein, Applicants respectfully request the Examiner to enter said amendments and respectfully submit that the application is in condition for allowance. Applicants reserve the right to file divisional application(s) on non-elected subject matter. The Commissioner is authorized to withdraw any necessary fees from Deposit Account 19-3880.

Respectfully submitted,



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Reg. No. 41,174Dated: October 4, 2006

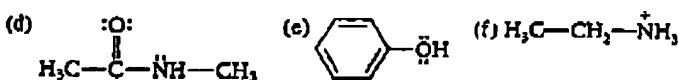
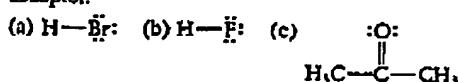
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primary alcohol, is a larger molecule than 1-butanol, its boiling point is the highest of the three. So far, the order of increasing boiling points is: pentane < 1-butanol < 1-hexanol. *tert*-butyl alcohol has about the same molecular mass as pentane, but the alcohol has a higher boiling point because of its polarity and hydrogen bonding. However, a *tert*-butyl alcohol molecule is more branched and more nearly spherical than the isomeric 1-butanol molecule; thus, the boiling point of *tert*-butyl alcohol should be lower than that of 1-butanol. Therefore, the correct order of boiling points is: pentane < *tert*-butyl alcohol < 1-butanol < 1-hexanol. (The respective boiling points in °C are 36, 82, 118, 157.)

PROBLEMS

8.11 Within each set, arrange the compounds in order of increasing boiling point.
(a) 4-ethylheptane, 2-bromopropane, 4-ethyloctane
(b) 1-butanol, 1-pentene, chloromethane

8.12 Label each of the following molecules as a hydrogen-bond acceptor, donor, or both. Indicate the hydrogen that is donated or the atom that serves as the hydrogen-bond acceptor.



3 8.4 SOLVENTS IN ORGANIC CHEMISTRY

A solvent is a liquid used to dissolve a compound. Solvents have tremendous practical importance. They affect the acidities and basicities of solutes. In some cases, the choice of a solvent can have dramatic effects on reaction rates. Understanding effects like these requires a classification of solvent types, to which Section 8.4A is devoted.

The rational choice of a solvent requires an understanding of solubility, that is, how well a given compound dissolves in a particular solvent. Section 8.4B discusses the principles that will allow you to make general predictions about the solubilities of organic compounds in different solvents. The effects of solvents on chemical reactions are closely tied to the principles of solubility.

Solubility is also important in biology. For example, the solubilities of drugs determine the forms in which they are marketed and used, and such important characteristics as whether they are absorbed from the gut and whether they pass from the bloodstream into the brain. Some of these ideas are explored in Section 8.5.

Because certain alcohols, alkyl halides, and ethers are among the most important organic solvents, this is a good point in your study of organic chemistry to study solvent properties.

A. Classification of Solvents

There are three broad solvent categories, and they are not mutually exclusive; that is, a solvent can be in more than one category.

1. A solvent can be *protic* or *aprotic*.
2. A solvent can be *polar* or *apolar*.
3. A solvent can be a *donor* or a *nondonor*.

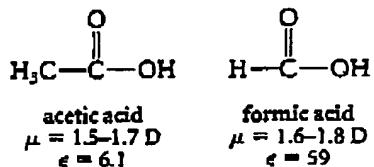
A **protic solvent** consists of molecules that can act as hydrogen-bond donors. Water, alcohols, and carboxylic acids are examples of protic solvents. Solvents that cannot act as hydrogen-bond donors are termed aprotic solvents. Ether, methylene chloride, and hexane are examples of aprotic solvents.

A **polar solvent** has a high dielectric constant; an **apolar solvent** has a low dielectric constant. The dielectric constant is defined by the *electrostatic law*, which gives the interaction energy E between two ions with respective charges q_1 and q_2 separated by a distance r :

$$E = k \frac{q_1 q_2}{\epsilon r} \quad (8.3)$$

In this equation, k is a proportionality constant and ϵ is the dielectric constant of the solvent in which the two ions are imbedded. This equation shows that when the dielectric constant ϵ is large, the magnitude of E , the energy of interaction between the ions, is small. This means that both attractions between ions of opposite charge and repulsions between ions of like charge are weak in a polar solvent. Thus, a polar solvent effectively separates, or shields, ions from one another. This means, in turn, that the tendency of oppositely charged ions to associate is less in a polar solvent than it is in an apolar solvent. If a solvent has a dielectric constant of about 15 or greater, it is considered to be polar. Water ($\epsilon = 78$), methanol ($\epsilon = 33$), and formic acid ($\epsilon = 59$) are polar solvents. Hexane ($\epsilon = 2$), ether ($\epsilon = 4$), and acetic acid ($\epsilon = 6$) are apolar solvents.

Unfortunately, the word *polar* has a double usage in organic chemistry. When we say that a *molecule* is polar, we mean that it has a significant dipole moment, μ (Sec. 1.2D). When we say that a *solvent* is polar, we mean that it has a high dielectric constant. In other words, solvent polarity, or dielectric constant, is a property of many molecules acting together, but molecular polarity, or dipole moment, is a property of individual molecules. Although it is true that all polar *solvents* consist of polar *molecules*, the converse is not true. The contrast between acetic acid and formic acid is particularly striking:



These two compounds contain identical functional groups and have very similar structures and dipole moments. Both are *polar molecules*. Yet they differ substantially in their dielectric constants and in their *solvent properties*! Formic acid is a polar solvent; acetic acid is not.

Donor solvents consist of molecules that can donate unshared electron pairs—that is, molecules that can act as Lewis bases. Ether, THF, and methanol are donor solvents. **Nondonor solvents** cannot act as Lewis bases; pentane and benzene are nondonor solvents.

Table 8.2 on p. 318 lists some common solvents used in organic chemistry along with their abbreviations and their classifications. This table shows that a solvent can have a combination of properties, as noted at the beginning of this section. For example, some polar solvents are protic (such as water and methanol), but others are aprotic (such as acetone).

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TABLE 8.2

Properties of Some Common Organic Solvents
(Listed in order of increasing dielectric constant)

Solvent	Structure	Common abbreviation	Boiling point, °C	Dielectric constant ^a	Class		
					Polar	Protic	Donor
hexane	<chem>CH3(CH2)4CH3</chem>	—	68.7	1.9			
1,4-dioxane [†]		—	101.3	2.2			x
carbon tetrachloride [‡]	<chem>CCl4</chem>	—	76.8	2.2			
benzene [†]		—	80.1	2.3			
diethyl ether	<chem>(C2H5)2O</chem>	Et ₂ O	34.6	4.3			x
chloroform	<chem>CHCl3</chem>	—	61.2	4.8			
ethyl acetate		EtOAc	77.1	6.0			x
acetic acid		HOAc	117.9	6.1		x	x
tetrahydrofuran		THF	66	7.6			x
methylene chloride	<chem>CH2Cl2</chem>	—	39.8	8.9			
acetone		Me ₂ CO, DMK	56.3	21	x		x
ethanol	<chem>C2H5OH</chem>	EtOH	78.3	25	x	x	x
hexamethylphosphoric triamide [†]	<chem>[(CH3)2N]3P(=O)</chem>	HMPA, HMPT	233	30	x		x
methanol	<chem>CH3OH</chem>	MeOH	64.7	33	x	x	x
nitromethane	<chem>CH3NO2</chem>	MeNO ₂	101.2	36	x		x
N,N-dimethylformamide		DMF	153.0	37	x		x
acetonitrile	<chem>CH3C≡N</chem>	MeCN	81.6	38	x		x
sulfolane		—	287 (dec)	49	x		x
dimethylsulfoxide		DMSO	189	47	x		x
formic acid		—	100.6	59	x	x	x
water	<chem>H2O</chem>	—	100.0	78	x	x	x
formamide		—	211 (dec)	111	x	x	x

^aMost values are at or near 25 °C[†]Known carcinogen[‡]Production banned in 1996.

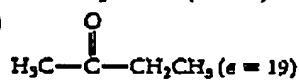
PROBLEM

8.13 Classify each of the following substances according to their solvent properties (as in Table 8.2).

(a) 2-methoxyethanol ($\epsilon = 17$)

(b) 2,2,2-trifluoroethanol ($\epsilon = 26$)

(c)



(d) 2,2,4-trimethylpentane ($\epsilon = 2$)

B. Solubility

One role of a solvent is simply to dissolve compounds of interest. Although finding a suitable solvent can involve some trial and error, certain principles can help us choose a solvent rationally. The discussion of solubility is divided into two parts: the solubility of covalent compounds, and the solubility of ionic compounds.

Solubility of Covalent Compounds In determining a solvent for a covalent compound, a useful rule of thumb is *like dissolves like*. That is, a good solvent usually has some of the molecular characteristics of the compound to be dissolved. For example, an apolar aprotic solvent is likely to be a good solvent for another apolar aprotic substance. In contrast, a protic solvent in which significant hydrogen-bonding interactions occur between molecules is likely to dissolve another protic substance in which hydrogen bonding between molecules is also an important cohesive interaction.

To illustrate, let's consider the water solubility of organic compounds. This is an important issue in biology, because water is the solvent in living systems. Consider the water solubility of the following compounds of comparable size and molecular mass:



water solubility:

virtually insoluble

soluble

miscible

Of these compounds, the alcohol, 1-propanol, is most soluble; in fact, it is miscible with water. This means that a solution is obtained when the alcohol is mixed with water in any proportion. Of the compounds shown, the alcohol is also most like water because it is protic. The ability to form hydrogen bonds with water is an important factor in water solubility. The ether contains an atom (oxygen) that can accept hydrogen bonds from water, although it cannot donate a hydrogen bond; hence, it has some water-like characteristics, but is less like water than the alcohol. Finally, the alkane (butane) and the alkyl halide (ethyl chloride) can neither donate nor accept hydrogen bonds and are therefore least like water; they are also the least soluble compounds on the list.

The same effect occurs in the following series:



water solubility:

miscible

7.7 mass %

0.58 mass %

Alcohols with long hydrocarbon chains, that is, large alkyl groups, are more like alkanes than are alcohols containing small alkyl groups. Because alkanes cannot form hydrogen bonds, they are insoluble in water, but they are soluble in other apolar aprotic solvents, including other alkanes. Hence, alcohols (as well as any other organic compounds) with long hydrocarbon chains are relatively insoluble in water and are more soluble in apolar aprotic solvents than alcohols with small alkyl chains.



✓ Study Guide Link 8.2
Boiling Points
and Solubilities



water solubility:

miscible

7.7 mass %

0.58 mass %



Study Guide Link 8.3
Solubility of Covalent
Compounds: A Deeper Look

Solvents consisting of polar molecules lie between the extremes of water on the one hand and hydrocarbons on the other. For example, consider the widely used solvent tetrahydrofuran (THF; see Table 8.2). Because THF can accept hydrogen bonds, it dissolves water and many alcohols. Because its dipole moment can interact favorably with other dipoles, it also dissolves polar compounds (for example, alkyl halides). On the other hand, because its hydrocarbon portion can take part in attractive van der Waals interactions, it also dissolves hydrocarbons. As a solvent for the reaction of a water-insoluble compound with water, THF is typically an excellent choice because it dissolves both compounds. For example, THF is the solvent of choice in oxymercuration of alkenes (Sec. 5.3A); it dissolves both water and alkenes.

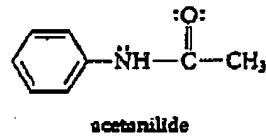
What you should begin to see from discussions of this sort are the *trends* to be expected in the solubility behavior of various compounds. You cannot be expected to remember absolute solubilities, but you should be able to make an intelligent guess about the relative solubilities of a given compound in different solvents or the relative solubilities of a series of compounds in a given solvent. This ability, for example, is required to solve the following problems.

PROBLEMS

8.14 In which of the following solvents should hexane be *least* soluble: diethyl ether, methylene chloride (CH_2Cl_2), ethanol, or 1-octanol? Explain.

8.15 (a) Into a separatory funnel is poured 200 mL of methylene chloride (density = 1.33 g/mL) and 55 mL of water. This mixture forms two layers. One milliliter of methanol is added to the mixture, which is then stoppered and shaken. Two layers are again formed. In which layer is the methanol likely to be dissolved? Explain.
 (b) The experiment is repeated, except that 1 mL of 1-nonanol is added instead of methanol. In which layer is the alcohol dissolved? Explain.

8.16 A widely used undergraduate experiment is the recrystallization of acetanilide from water. Acetanilide (see following structure) is moderately soluble in hot water, but much less soluble in cold water. Identify one structural feature of the acetanilide molecule that would be expected to contribute positively to its solubility in water and one that would be expected to contribute negatively.



Solubility of Ionic Compounds Because of the importance of both ionic reagents and ionic reactive intermediates in organic chemistry, the solubility of ionic compounds is worth special attention. Ionic compounds in solution can exist in several forms, two of which, *ion pairs* and *dissociated ions*, are shown in Fig. 8.2. In an ion pair, each ion is closely associated with an ion of opposite charge. In contrast, dissociated ions move more or less independently in solution and are surrounded by several solvent molecules, called collectively the *solvation shell* or *solvent cage* of the ion. Solvation refers to the favorable interaction of a dissolved molecule with solvent. When solvent molecules interact favorably with an ion, they are said to *solvate* the ion.

Ion separation and ion solvation are mechanisms by which ions are stabilized in solution. If you think of the ion dissolution sequence in Fig. 8.2 as an ordinary chemical

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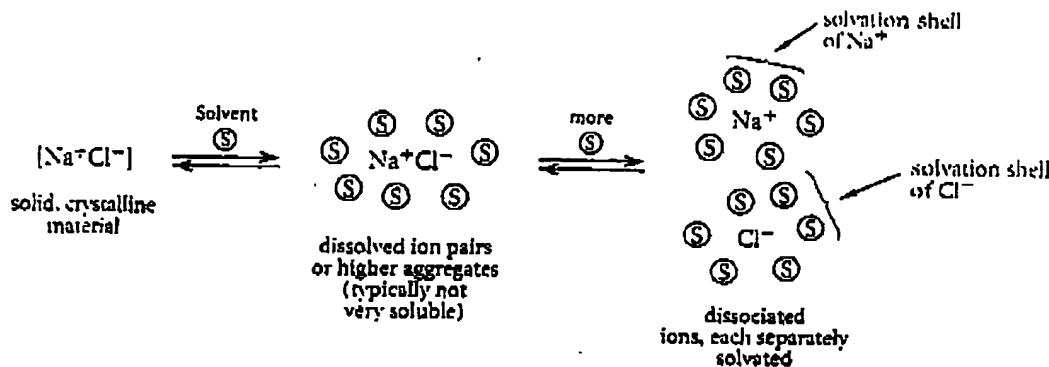
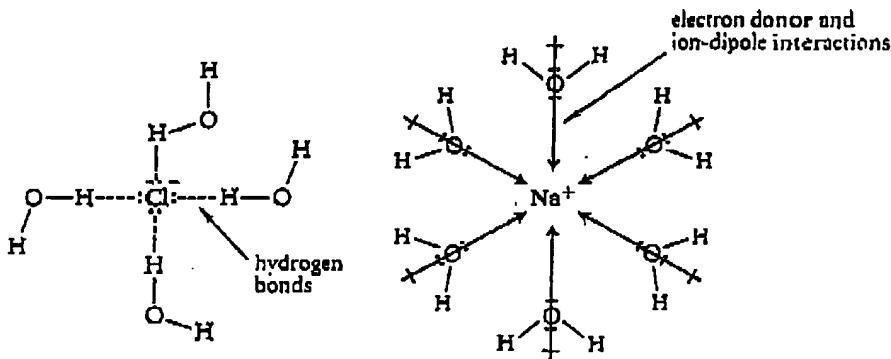


FIGURE B.2 Ions in solution can exist as ion pairs and dissociated ions. The solubility of an ionic compound depends on the ability of the solvent to break the electrostatic attractions between ions and form separate solvation shells around the dissociated ions. (The colored circles are solvent molecules.)

equilibrium, you can see that anything that favors the right side of this equilibrium tends to make ions soluble. The separation and solvation of ions reduce the tendency of the ions to associate into aggregates and ultimately, to precipitate as solids from solution. Hence, ionic compounds are relatively soluble in solvents in which ions are well separated and solvated. What solvent properties contribute to the separation and solvation of ions?

The ability of a solvent to *separate* ions is measured by its dielectric constant ϵ in Eq. 8.3 on p. 317. Look carefully at this equation again. The energy of attraction of two ions of opposite charge is reduced in a solvent with a high dielectric constant. Hence, ions of opposite charge in solvents with high dielectric constants have a reduced tendency to associate, and thus a greater solubility.

Solvent molecules *solvate* ions in several ways, illustrated in the following structures for the solvation of sodium and chloride ions by water molecules:



Anions, such as the chloride ion, are solvated by *hydrogen bonding*: that is, they accept hydrogen bonds from the solvent. Cations, such as the sodium ion, are solvated by what are called collectively *donor interactions*. In one type of donor interaction, an atom with an unshared electron pair on a solvent molecule (such as the oxygen of water) acts as a Lewis base (electron donor) toward an electron-deficient cation. In the second type of donor interaction,

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a solvent molecule aligns itself so that the negative end of its dipole moment vector is pointed toward the cation. This type of interaction is called an *ion-dipole attraction*.

To summarize: Three solvent properties contribute to the solubility of ionic compounds: *polarity* (high dielectric constant), by which solvent molecules separate ions of opposite charge; *proticity* (hydrogen-bond donor capability), by which solvent molecules solvate anions; and *electron-donor ability*, by which solvent molecules solvate cations through Lewis-base and ion-dipole interactions. It follows, then, that *the best solvents for dissolving ionic compounds are polar, protic, donor solvents*.

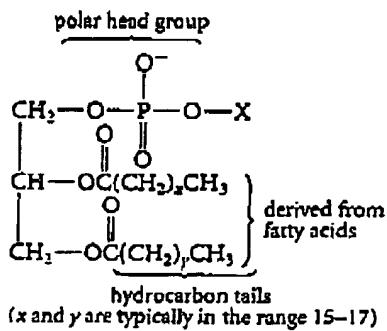
Thus, water is the ideal solvent for ionic compounds, something you probably know from experience. First, because it is polar—it has a very large dielectric constant—it is effective in separating ions of opposite charge. Second, because it is a donor solvent—a good Lewis base—it readily solvates cations. Finally, because it is protic—a good hydrogen-bond donor—it readily solvates anions. In contrast, hydrocarbons such as hexane do not dissolve ordinary ionic compounds because such solvents are apolar, aprotic, and nondonor solvents. Some ionic compounds, however, have appreciable solubilities in *polar aprotic* solvents such as acetone or DMSO (see Table 8.2). Although these solvents lack the protic character that solvates anions, their donor capacity solvates cations and their polarity separates ions of opposite charge. However, it is not surprising that because polar aprotic solvents lack the protic character that stabilizes anions, most salts are less soluble in these solvents than in water, and salts dissolved in polar aprotic solvents exist to a greater extent as ion pairs (see Fig. 8.2).

8.5 APPLICATIONS OF SOLUBILITY AND SOLVATION PRINCIPLES

A. Cell Membranes and Drug Solubility

Solubility is a crucial issue in drug action. If a drug is to be administered in an aqueous solution, it must have adequate aqueous solubility. However, water solubility is not the whole story. For drugs to act, they must get to their sites of action. For many drugs, this means that they must enter cells. The only way for a drug to get into a cell is for it to pass through the *cell membrane*, the "envelope" that surrounds the cell. Drugs and other substances pass through cell membranes by a variety of mechanisms; in some cases, transport requires carrier molecules imbedded in the membrane; and, in some cases, transport requires the expenditure of metabolic energy. However, in many cases, drugs simply pass unassisted through the cell membrane. It turns out that the ability of a molecule to penetrate a cell membrane is very much a solubility issue. To understand this, let's examine the structure of a cell membrane.

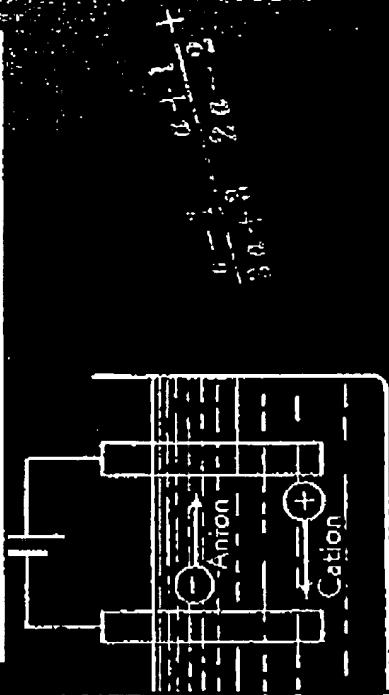
Cell membranes contain a high concentration of molecules called *phospholipids*. The general structure of a phospholipid and two specific examples are as follows:



Examples:	
—X	Name of phospholipid
—CH ₂ CH ₂ N ⁺ (CH ₃) ₃	phosphatidylcholine (lecithin)
—CH ₂ CH ₂ NH ₃ ⁺	phosphatidylethanolamine

OXFORD
PAPERBACK REFERENCE

CHEMISTRY



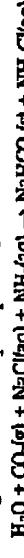
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1996

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solvent

sodium carbonate from calcium carbonate and sodium chloride. The calcium carbonate is first heated to give calcium oxide and carbon dioxide, which is bubbled into a solution of sodium chloride in ammonia. Sodium hydrogentarbonate is precipitated:



The sodium hydrogentarbonate is heated to give sodium carbonate and carbon dioxide. The ammonium chloride is heated with calcium oxide (from the first stage) to regenerate the ammonia. The process was patented in 1861 by the Belgian chemist Ernest Solvay (1838-1922).

solvent A liquid that dissolves another substance or substances to form a solution. Polar solvents are compounds such as water and liquid ammonia, which have dipole moments and consequently high dielectric constants. These solvents are capable of dissolving ionic compounds or covalent compounds that ionize (see solvation). Nonpolar solvents are compounds such as ethoxyethane and benzene, which do not have permanent dipole moments. These do not dissolve ionic compounds but will dissolve nonpolar covalent compounds. Solvents can be further categorized according to their proton-donating and accepting properties. Aprotic protic solvents self-ionize and can therefore act both as proton donors and acceptors. A typical example is water:



Aprotic solvents neither accept nor donate protons; tetrachloromethane (carbon tetrachloride) is an example.

solvent extraction The process of separating one constituent from a mixture by dissolving it in a solvent in which it is soluble but in which the other constituents of the mixture are not. The process is usually carried out in the liquid phase, in which case it is also known as liquid-liquid extraction. In liquid-liquid extraction, the solution containing the desired constituent must be immiscible with the rest of the mixture. The process is widely used in extracting oil from oil-bearing materials.

solvolysis A reaction between a compound and its solvent. See hydrolysis, sonochemistry.

sonochemistry The study of chemical reactions in liquids subjected to high-intensity sound or ultrasound. This causes the formation, growth, and collapse of tiny bubbles within the liquid, generating localized centres of very high temperature and pressure, with extremely rapid cooling rates. Such conditions are suitable for studying novel reactions, decomposing polymers, and producing amorphous materials.

sorption * Absorption of a gas by a solid.

sorption pump A type of vacuum pump in which gas is removed from a system by absorption on a solid (e.g. activated charcoal or a zeolite) at low temperature.

species A chemical entity, such as a particular atom, ion, or molecule.

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spectroscopic

specific 1. Denoting that an extensive physical quantity so described is expressed per unit mass. For example, the specific latent heat of a body is its latent heat per unit mass. When the extensive physical quantity is denoted by a capital letter (e.g. *I* for latent heat), the specific quantity is denoted by the corresponding lower-case letter (e.g. *i* for specific latent heat). **2.** In some older physical quantities the adjective 'specific' was added for other reasons (e.g. specific gravity, specific resistance). These names are now no longer used.

specific activity See activity.

specific gravity See relative density; specific

specific heat capacity See heat capacity.

spectrochemical series A series of ligands arranged in the order in which they cause splitting of the energy levels of *d*-orbitals in metal complexes (see crystal-field theory). The series for some common ligands has the form:



spectrograph See spectroscope.

spectrometer Any of various instruments for producing a spectrum and measuring the wavelengths, energies, etc. involved. A simple type, for visible radiation, is a spectroscope equipped with a calibrated scale allowing wavelengths to be read off or calculated. In the X-ray to infrared region of the electromagnetic spectrum, the spectrum is produced by dispersing the radiation with a prism or diffraction grating (or crystal). In the case of hard X-rays, some form of photoelectric detection is used, and the spectrum can be obtained as a graphical plot, which shows how the intensity of the radiation varies with wavelength. Such instruments are also called spectrophotometers. Spectrometers also exist for investigating the gamma-ray region and the microwave and radio-wave regions of the spectrum (see electron-spin resonance; nuclear magnetic resonance). Instruments for obtaining spectra of particle beams are also called spectrometers (see spectrum; platolectron spectroscopy).

spectrophotometer See spectrometer.

spectroscope An optical instrument that produces a spectrum for visual observation. The first such instrument was made by R. W. Bunsen; in its simplest form it consists of a hollow tube with a slit at one end by which the light enters and a collimating lens at the other end to produce a parallel beam, a prism to disperse the light, and a telescope for viewing the spectrum. In the spectrograph, the spectroscope is provided with a camera to record the spectrum.

For a broad range of spectroscopic work, from the ultraviolet to the infrared, a diffraction grating is used instead of a prism. See also spectrometer.